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INTELLECTUAL  
PROPERTY INDIA

GOVERNMENT OF INDIA  
MINISTRY OF COMMERCE & INDUSTRY,  
PATENT OFFICE, DELHI BRANCH,  
W - 5, WEST PATEL NAGAR,  
NEW DELHI - 110 008.

I, the undersigned, being an officer duly authorized in accordance with the provision of the Patent Act, 1970 hereby certify that annexed hereto is the true copy of the Application and Complete Specification and Drawing Sheets filed in connection with Application for Patent No.844/Del/02 dated 14<sup>th</sup> August 2002.

Witness my hand this 12<sup>th</sup> Day of May 2003.

(S.K. PANGASA)

Assistant Controller of Patents & Designs

**PRIORITY**

**DOCUMENT**

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**FORM-1** 14 AUG 2002  
**THE PATENTS ACT, 1970**  
(39 of 1970)  
**APPLICATION FOR GRANT OF A PATENT**  
(See section 5(2), 7, 54 and 135 and rules 39A)

India Patent Office  
New Delhi  
Received Rs. 5080/- in cash.  
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Vide Entry No. 2500 in the Register of Valuables  
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I,

- (a) DR. Y. S. PARMAR UNIVERSITY OF HORTICULTURE AND FORESTRY,
- (b) Nauni, Solan, Himachal Pradesh,
- (c) An Indian University.

2. I hereby declare -

- (a) that we are in possession of an invention "A process for the estimation of volatile substances".
- (b) that the complete specification relating to this invention is filed with this application.
- (c) that there is no lawful ground of objection to the grant of a patent to us.

3. I further declare that the inventor for the said invention are :

- (a) AMIT NATH, S.K. PATYAL & J.K. DUBEY
- (b) Department of Entomology and Apiculture, Dr. Y.S. Parmar University of Horticulture and Forestry, Nauni, Solan (H.P.)
- (c) All Indian Nationals.

4. I claim the priority from the application (s) filed in convention countries, particulars of which are as follows:

- (a) .....NIL.....

5. I state that the said invention is an improvement in or modification of the invention the particulars of which are as follows and of which I am the applicant/patentee:

- a) .....NIL.....

6. I state that the application is divided out of my application, the particulars of which are given below and pray that this application deemed to have been filed on .....NA..... under section 16 of the Act.

- (a) .....NIL.....

7. That we are the assignee or legal representatives of the true and first inventors.

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**THE PATENTS ACT, 1970**

COMPLETE

**SPECIFICATION**

**SECTION 10**

**TITLE**

"A process for the estimation of volatile substances".

**APPLICANT**

DR. Y. S. PARMAR UNIVERSITY OF HORTICULTURE AND  
FORESTRY, Nauni, Solan, Himachal Pradesh, an Indian  
University. INDIA.

The following specification particularly describes and ascertains the nature of this  
invention and the manner in which is to be performed

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Another method known in the art, is the headspace method and in which vapours are drawn from the headspace of a septum-sealed vial containing a sample to be analysed. The vial was then heated to drive out dissolved organics out of solution and into the vapour headspace (Ward; Clydie, US Patent Application no. 301385 dated April 27, 1999; US patent: 6,286,375).

In another method, the vial containing volatile sample was heated by Green et al 2000 and agitated to enhance a transport rate of the volatile sample from material to the headspace of the vial (United States patent 6,146,895, November 14, 2000).

For extraction Ray, et al., 1997 proposed extractor having sample chamber pressurizable either by gas or mechanical means. The sample chamber was construed with removable liner of poly tetrafluorethylene (United States Patent 5,607,234 March 4, 1997).

Augenblick et al. 1994 describes yet another method and apparatus for collecting gases from the sample headspace of sealed container. Gas sample from the headspace to instrument was carried by carrier gas (United State Patent no. 5,363,707 dated November 15, 1994). Vibration used to promote the formation of sample gas.

Another object of this invention is to propose a process for the estimation of volatile substances, which obviates the disadvantages associated with those of the prior art.

Yet another object of this invention is to propose a process for the estimation of volatile substances and wherein chloroform residues are released from the sample into headspace under partial vacuum or at low pressure whereby reducing risk of leakage of volatile substances.

Still another object of this invention is to propose a process for the estimation of volatile substances and wherein no additional carrier gas is required.

A further object of this invention is to propose a process for the estimation of volatile substances and wherein prolonged high temperature is not required.

A still further object of this invention is to propose a process for the estimation of volatile substances, which involves low cost of apparatus.

Thereafter, the flask is removed from the heat source and the sample to be analyzed is introduced into said flask and then maintained at a second temperature lower than said first temperature. However, after introducing the sample into said flask, the flask is closed. As the flask is in a closed status and the temperature reduced from a first to a second temperature, a vacuum or low pressure is created within said flask. By way of example and without implying any limitation thereto, the first temperature is the boiling temperature and the second temperature is approximately 45°C.

The flask is maintained at the second temperature for approximately 45 minutes. Thereafter, the vacuum is broken by purging air into the flask. Turbulence is caused within said vessel so as to disperse the volatile substances in the head space within the closed flask. A sample is removed from the headspace and analyzed.

Preferably the sample is wrapped in a foil and introduced into the flask.

Thus according to one aspect of the present invention there is closed container containing sample/volatile material and water. Sample is added prior to closure of the container.

Examples of closed containers which may be useful in accordance with invention include glass flask (250 ml) with B-24 neck, this flask is attached with 7 cm stopper having B-24 joint at one end and 1.5 cm diameter hole at the top closed by a 2 cm silicon cork.

The volatile substance may in general comprise of flavours present in food, beverages and other additives. However, other volatile contaminants like halogens can be estimated too.

Boiling of water prior to adding sample expel the air inside the flask and when temperature of flask is lowered vacuum is created which causes the release of volatile substances from the plant matrix. Thus the processes involving introduction of plant matrix/sample into the boiled water and equilibrating at a constant temperature followed by introduction of air, is the point of completion of the treatment.

#### **Working examples:**

##### **Example-I**

Weigh 1 g crop material into a aluminium foil and fold loosely in a packet form so that crop material could easily come in contact with the water, after its insertion into the flask. First take thirty milliliter distilled water in 250 ml flask, boil then replace flask from the heat source, push in the packet containing the material and immediately encap with stopper (stopper plugged with silicon cork). Swirl the flask until material from the aluminium foil come in the contact with the warm water. Equilibrate the flask at 40°C for one hour in the incubator. At the end, insert needle of 10 ml syringe through the silicon cork and introduce air into it for 30 seconds. Replace the plunger, move plunger up and down three times. Then suck 10 ml vapours from the flask into the syringe and then take out the syringe. Out of 10 ml vapours expel 9 ml and inject remaining vapours into gas chromatograph fitted with ECD and capillary column for qualitative and quantitative estimation.

respectively. Under these conditions minimum detection limit for chloroform is 5 ppb.

#### DESCRIPTION WITH REFERENCE TO DRAWINGS

Further objects and advantages of this invention will be more apparent from the ensuing description when read in conjunction with the accompanying drawing, which illustrates an exploded view of the flask of the present invention.

The flask 1 has a neck 2. A stopper 3 is adapted to close the mouth 4 of flask 1. Stopper 3 has a lower section 5, which is a conical member and an upper frusto conical section 6. Mouth 7 is adapted to be closed by a closure 8.



5. A process as claimed in claim 1 wherein the heating of water is carried out at atmospheric pressure.
6. A process for the estimation of volatile substances substantially as herein described and illustrated.
7. An apparatus for the estimation of volatile substances comprising a flask having a stopper adapted to fit and close the mouth of said flask, a closure member for closing the mouth of said stopper.
8. An apparatus as claimed in claim 7 wherein said stopper is a hollow member.
9. An apparatus as claimed in claim 7 wherein said stopper comprises a lower section of a conical section and an upper section of a frusto conical section.
10. An apparatus for the estimation of volatile substances substantially as herein described and illustrated.

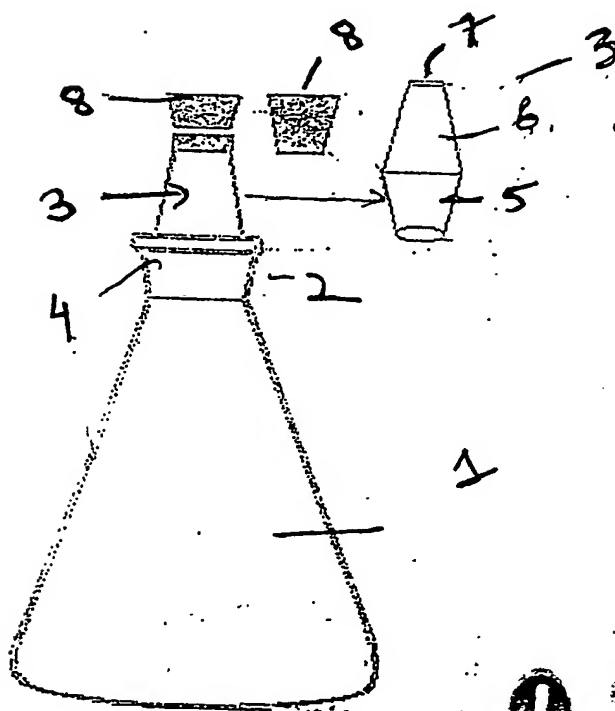
DATED THIS 13<sup>th</sup> DAY OF AUGUST, 2002.



( G.S.DAVAR )  
OF L.S.DAVAR & CO.,  
APPLICANTS ATTORNEY

APPLICANT: DR. Y. S. PARMAR UNIVERSITY OF  
HORTICULTURE AND FORESTRY

TOTAL SHEETS 1  
SHEET NO. 1



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APPLICANTS ATTORNEY

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